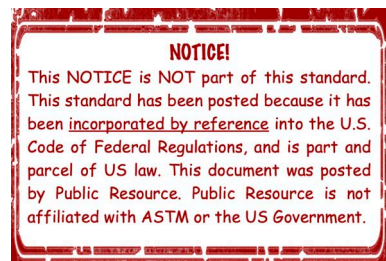


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Designation: D5198 – 09

Standard Practice for Nitric Acid Digestion of Solid Waste¹

This standard is issued under the fixed designation D5198; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.



1. Scope

1.1 This practice describes the digestion of solid waste using nitric acid for the subsequent determination of inorganic constituents by plasma emission spectroscopy or atomic absorption spectroscopy.

1.2 The following elements may be solubilized by this practice:

aluminum	manganese
beryllium	mercury
cadmium	nickel
chromium	phosphorus
copper	vanadium
iron	zinc
lead	

1.3 This practice is to be used when the concentrations of total recoverable elements are to be determined from a waste sample. Total recoverable elements may or may not be equivalent to total elements, depending on the element sought and the sample matrix. Recovery from refractory sample matrices, such as soils, is usually significantly less than total concentrations of the elements present.

NOTE 1—This practice has been used successfully for oily sludges and a municipal digested sludge standard [Environmental Protection Agency (EPA) Sample No. 397]. The practice may be applicable to some elements not listed above, such as arsenic, barium, selenium, cobalt, magnesium, and calcium. Refractory elements such as silicon, silver, and titanium, as well as organo-mercury are not solubilized by this practice.

1.4 This practice has been divided into two methods, A and B, to account for the advent of digestion blocks. Method A utilizes an electric hot plate; Method B utilizes an electric digestion block.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appro-*

priate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 7.

2. Referenced Documents

- 2.1 *ASTM Standards:*²
D1193 Specification for Reagent Water

3. Summary of Practice

3.1 A weighed portion of the waste sample is mixed with 1 + 1 nitric acid (HNO₃) in an Erlenmeyer flask. The flask is heated for 2 h at 90 to 95°C to dissolve the elements of interest. After cooling, the contents of the flask are diluted with reagent water and filtered, and the filtrate is made up to appropriate volume for subsequent analysis.

4. Significance and Use

4.1 A knowledge of the inorganic composition of a waste is often required for the selection of appropriate waste disposal practices. Solid waste may exist in a variety of forms and contain a range of organic and inorganic constituents. This practice describes a digestion procedure which dissolves many of the toxic inorganic constituents and produces a solution suitable for determination by such techniques as atomic absorption spectroscopy, atomic emission spectroscopy, and so forth. The relatively large sample size aids representative sampling of heterogeneous wastes. The relatively small dilution factor allows lower detection limits than most other sample digestion methods. Volatile metals, such as lead and mercury, are not lost during this digestion procedure, however organo-lead and organo-mercury may not be completely digested. Hydride-forming elements, such as arsenic and selenium, may be partially lost. Samples with total metal contents greater than 5 % may give low results. The analyst is responsible for determining whether this practice is applicable to the solid waste being tested.

¹ This practice is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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METHOD A – HOT PLATE

5. Apparatus

- 5.1 *Analytical Balance*, capable of weighing to 0.01 g.
- 5.2 *Erlenmeyer Flasks*, 125 mL.
- 5.3 *Graduated Cylinder*, 50 mL.
- 5.4 *Electric Hot Plate*, adjustable, capable of maintaining a temperature of 90 to 95°C.
- 5.5 *Watch Glasses*.
- 5.6 *Thermometer*.
- 5.7 *Funnels*, glass or plastic.
- 5.8 *Volumetric Flasks*, glass-stoppered, 200 mL.
- 5.9 *Filter Paper*, quantitative, medium flow rate, Whatman No. 40 or equivalent.
- 5.10 *Fume Hood*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type II of Specification D1193.

6.3 *Nitric Acid*, concentrated, reagent grade.

6.4 *Nitric Acid (1+1)*—Add slowly, with stirring, 200 mL of concentrated nitric acid (HNO₃, sp gr 1.42) to 200 mL water. Cool the mixture and store in a clean pint glass bottle.

³ *Reagent Chemicals, American Chemical Society Specifications*, Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagent Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia*.

7. Hazards

7.1 Add the nitric acid mixture slowly, with swirling, to the sample. Samples containing carbonates may foam excessively during acid addition and result in loss of sample. Nitric acid may react violently with some samples containing organic material.

7.2 Addition of acid and sample digestion must be conducted in a hood with adequate ventilation and shielding to avoid contact with nitrogen oxides, acid fumes, or toxic gases.

8. Procedure

8.1 Weigh 5 g of a thoroughly mixed waste sample to the nearest 0.01 g into a tared Erlenmeyer flask.

8.2 With a graduated cylinder, slowly add 25 mL of 1+1 nitric acid to the flask. Swirl the flask to wet the sample completely.

8.3 Carry a blank of 25 mL of 1+1 nitric acid through the procedure.

8.4 Place the flask on a cold hot plate, cover with a watch glass, and set the hot plate to maintain a temperature of 90 to 95°C.

8.5 Heat the flask and contents for 2 h, occasionally swirling the flask to wash down any sample adhering to the walls. Check the solution temperature with the thermometer and adjust the heat if necessary.

8.6 After 2 h, remove the flask from the hot plate and cool to room temperature. Add 50 mL of reagent water to the flask, washing down the flask walls during addition. Swirl the flask to mix the contents.

8.7 Filter the contents of the flask into a 200 mL volumetric flask. Rinse the flask and filter paper with several small portions of reagent water and add the rinsings to the volumetric flask.

8.8 Dilute the solution in the volumetric flask to the mark with reagent water and mix thoroughly. The solution is now ready for analysis.

METHOD B – DIGESTION BLOCK

9. Apparatus

- 9.1 *Analytical Balance*, capable of weighing to 0.01 g.
- 9.2 *Fume Hood*.
- 9.3 *Graduated Digestion Tubes*.
- 9.4 *Graduated Cylinder*, 50 mL.
- 9.5 *Digestion Tube Filters*.
- 9.6 *Digestion Block*, adjustable, capable of maintaining a temperature of 90 to 95°C.

10. Reagents

10.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type II of Specification **D1193**.

10.3 *Nitric Acid*, concentrated, reagent grade.

10.4 *Nitric Acid (1 + 1)*—Add slowly, with stirring, 200 mL of concentrated nitric acid (HNO₃, sp gr 1.42) to 200 mL water. Cool the mixture and store in a clean pint glass bottle.

11. Hazards

11.1 Add the nitric acid mixture slowly, with swirling, to the sample. Samples containing carbonites may foam excessively during acid addition and result in loss of sample. Nitric acid may react violently with some samples containing organic material.

11.2 Addition of acid and sample digestion must be conducted in a hood with adequate ventilation and shielding to avoid contact with nitrogen oxides, acid fumes, or toxic gases.

12. Procedure

12.1 Weigh 5 g of a thoroughly mixed waste sample to the nearest 0.01 g and transfer into a graduated digestion tube.

12.2 With a graduated cylinder, slowly add 25 mL of 1 + 1 nitric acid to the digestion tube. Swirl the tube to wet the sample completely.

12.3 Carry a blank of 25 mL of 1 + 1 nitric acid through the procedure.

12.4 Place the digestion tube in a digestion block in a fume hood and set the digestion block to maintain a temperature of 90 to 95°C.

12.5 Heat the tube and contents for 2 h, until a volume of approximately 15 mL remains.

12.6 After 2 h, remove the tube from the digestion block and cool to room temperature.

12.7 Filter the contents of the tube with a digestion tube filter.

12.8 Dilute the solution to 50 mL. The solution is now ready for analysis.

13. Precision and Bias

13.1 No statement is made about either the precision or bias since this practice does not produce a test result. **Appendix X1** contains representative results obtained with this practice (Method A) and subsequent analysis.

14. Keywords

14.1 digestion; nitric acid; waste

APPENDIX

(Nonmandatory Information)

X1. REPRESENTATIVE ANALYSES OF SAMPLES AFTER NITRIC ACID DIGESTION

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TABLE X1.1 Nitric Acid Digestion—Analysis of Standard Sludge Sample (EPA Sample No. 397)

Element	Concentration, mg/kg		Percent Recovery
	Actual	Found	
Zn	1323	1300	98
Mn	205	235	115
Pb	519	500	96
Cd	20.8	24.1	116
Cr	204	218	107
Fe	16 155	16 400	102
V	13.0	12.7	98
Cu	1095	1130	103
Ni	198	186	94
Al	4558	4500	99
Ti	2121	95	4.5
P	11 573	11 800	102
Be	0.28	0.5	179
As	17	9	53
Ag	81	7.4	9.1
Hg	16.3	13.6	83

TABLE X1.2 Nitric Acid Digestion—Replicate Analysis of Used Motor Oil, mg/kg

Element	Run 1	Run 2	Run 3
Zn	1170	1150	1170
Mn	2.5	2.1	2.1
Pb	43	40	35
Cd	0.6	0.5	0.4
Cr	2.8	2.1	2.3
Fe	74	66	66
V	1.32	1.04	0.92
Cu	3.8	3.6	3.6
Ni	0.7 ND ^A	0.7 ND	0.7 ND
Ba	0.6	0.5	0.5
Be	0.04 ND	0.04 ND	0.04 ND
Hg	0.08 ND	0.08 ND	0.08 ND

^A ND = not detected at the detection limit shown.

TABLE X1.3 Nitric Acid Digestion—Recovery of Used Motor Oil Matrix Spikes

Element	mg/kg		Percent Recovery
	Added	Found	
Mn	3.2	3.3	103
Pb	3.4	3.0	88
Cd	2.0	1.6	80
Cr	2.0	2.4	120
V	2.6	1.0	38
Cu	2.2	4.2	191
Ni	2.8	2.75	98
Ba	2.3	2.1	91
Be	1.6	1.3	81
Hg	0.08	0.09	113

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